

RESIDUES OF LINDANE AND CHLORPYRIFOS IN FIREWOOD AND WOODSMOKE

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ABSTRACT

Pine bark beetle insecticide treatment plots were established on the Ocala National Forest, in central Florida. Each plot consisted of five sand pine, *Pinus clausa* (Chapm. Ex. Engelm.) Vasey ex. Sarg., trees treated with either 0.5% lindane (benzene hexachloride) or 2% chlorpyrifos (O,O-diethyl 0-(3,5,6-trichloro-2-pyridyl) phosphorothioate). After 4 months, mean residue levels ranged from 0.32 to 35.8 mg/kg for lindane and < 0.1 to 76.1 mg/kg for chlorpyrifos. Chlorpyrifos was more persistent in wood than lindane. In a separate laboratory study, lindane or chlorpyrifos were applied to powdered wood and then burned under controlled conditions to determine carryover in combustion products. With slow heating to 500°C (20°C/min.), 42.7% of the lindane and 28.3% of the chlorpyrifos were recovered in the smoke stream. With rapid combustion at 600°C, all lindane and chlorpyrifos residues were thermally degraded. These findings were related to the risk of burning insecticide treated wood as firewood inside houses. Even under the worst case slow burning conditions, human exposure to airborne residues would be well under "safe-sided" threshold limit values and less than 0.2% of the acceptable daily intake established by the World Health Organization.

Key Words: Insecticide residues, lindane, chlorpyrifos, thermal degradation.

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INTRODUCTION

Bark beetles, especially the southern pine beetle, *Dendroctonus frontalis* Zimmerman, are a serious threat to forest stands and individual high value trees throughout the South (Thatcher 1978). Southern pine beetle caused mortality of trees in yards of private homes and on city streets often resulting in substantial losses through removal costs, losses in aesthetic values, and devaluation of property (Berisford et al. 1981b). Insecticidal control of bark beetle infestations in high value urban trees is an effective technique. However, the disposal of insecticide treated trees is sometimes a problem. In some cases, these treated trees may be used for firewood. Since firewood was not considered as an end use of insecticide-treated trees, most registered compounds were not evaluated during the registration process for their human-exposure potential when burned. With increased usage of insecticide-treated wood, questions have been raised regarding residue fate and the potential for human exposure.

We report here a study on lindane and chlorpyrifos residues in trees which could be used for firewood and the fate of those residues when burned under

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controlled combustion conditions. Implications on the suitability of lindane or chlorpyrifos treated pines for firewood and potential human exposure to residues are discussed.

MATERIALS AND METHODS

The insecticide treatment plots were established in the Lake George Ranger District, Ocala National Forest, FL, (see Cantrell 1984 for complete site description). Each plot consisted of 5 sand pine, *Pinus clausa* (Chapm. ex. Engelm.) Vasey ex. Sarg., trees. Trees were numbered with aluminum tags and three diameter at breast height (DBH) was measured prior to insecticide treatment. Each tree was sprayed with a 0.5% lindane (20% EC) or 2% chlorpyrifos (Dursban 4E)⁵ solution. Insecticides were applied in accordance with label directions using back pack sprayers to thoroughly cover the trunk from the lower crown to ground level.

Firewood Sample Processing

Five replications of the two insecticide treatments were individually sampled 4, 8, and 12 months after application in 1982. Composites of bark and wood tissue from the base of each sampled tree were obtained using a series of close-spaced chain saw cuts. Subsamples were kept separate for moisture determination. Kerf samples taken from the bottom 30 cm of each trunk were further reduced by grinding in a Wiley mill until they were small enough to filter through a 1 mm mesh screen. Samples were then frozen at -20°C until analyzed.

Residue Extraction Analyses

Residue samples for lindane and chlorpyrifos determination were soxhlet extracted and residue levels were quantitated by gas-liquid chromatography. A 30 g sample was weighed into a soxhlet thimble and extracted with 250 ml of acetone for 12 hours. The extracts were further cleaned up by liquid-liquid partitioning.

Samples were analyzed for lindane using a Tracor Model 222 gas chromatograph equipped with a Ni63 electron capture detector and a 2 M X 4 mm I.D. glass column packed with 3% OV-1 on Chromosorb WHP. The detector, inlet, and column temperatures were 350, 250, and 200°C, respectively. The carrier and purge gases were 5% methane/95% argon, with a flow rate of 45 ml/min and 10 ml/min, respectively.

Chlorpyrifos analysis was conducted with a Tracor Model 550 gas chromatograph equipped with a flame photometric detector and a glass column as described above. The hydrogen and air flow were optimized for maximum response and the detector temperature was 220°C. The carrier gas was nitrogen at a flow rate of 40 ml/min.

Residue levels were determined by comparison of peak height in sample chromatograms to those of analytical standards obtained from US Environmental Protection Agency, Research Triangle Park, NC. Percent recovery values were used to correct sample concentrations. **Lindane** and chlorpyrifos percent recoveries

⁵ The use of trade, firm, or corporation names in this publication is for the information and convenience of the reader. Such use does not constitute an official endorsement of approval by the U. S. Department of Agriculture of any product to the exclusion of others that may be suitable.

(\pm one standard deviation, $n = 5$) from fortified wood samples were 97.5 ± 3.5 and 88.5 ± 6.0 , respectively.

Combustion Procedures

A key factor in conducting this study was to select a combustion procedure that was reproducible and that would simulate the wide range of conditions under which insecticide treated wood could be burned. It is well known, for example, that wood burns differently in fireplaces than in wood stoves, or under conditions of flaming and smoldering combustion. A horizontal tube furnace procedure was selected for this study. The tube furnace (Lindberg Model 54451) was fitted with a 5 mm diameter 1.2 m long quartz tube and other special fittings and sampling apparatus (McMahon et al. 1985). This system allowed small (0.5 g) fuel samples to be burned under preselected combustion conditions and flow rates. It also permitted quantitative sampling of combustion gases and particles generated during each burn. The quantity of particulate matter released was used as an indication of flaming (rapid) combustion or smoldering (slow) combustion. Preliminary experiments indicated that smoldering conditions could be simulated by slowly heating the sample ($20^{\circ}\text{C}/\text{min}$) to 500°C . Flaming conditions were simulated by inserting the fuel sample directly into the furnace after it had been preheated to 500°C . During some of the runs, it was found that insertion at 500°C was a marginal flaming condition. Thus, some repeat runs were made at 600°C which proved successful as a simulation for rapid flaming combustion.

Swamp chestnut oak, *Quercus michauxii* Nutt., was used as a representative wood fuel matrix in the combustion study. The choice of a hardwood species for this work was based on an extension of a study already underway which had examined the release of herbicides from stem-injected hardwoods (McMahon et al. 1985). Although many types of wood are burned in wood stoves and fireplaces, it has been shown that the way a wood-burning appliance is operated (i.e., slow or rapid burning) has a much greater influence on the products of combustion than wood species or moisture content (Shelton and McGrath 1981; Ayer 1981). These findings influenced the selection of burning parameters for the tube furnace described earlier.

Pieces of oven dried branch wood were ground in a Wiley mill to pass through a 20-mesh screen. Small samples of the powdered wood (0.5 g) were treated with a designated level of insecticide. Application rates between 380 and 514 μg of insecticide per gram of wood were used. These levels were selected to represent a worst case situation and to insure positive analytical results in the smoke samples. Combustion gases, insecticide residues, and particulate matter were collected on a glass transfer tube, glass fiber filter, and polyurethane foam (PUF) plug system described by McMahon et al. (1985). Components of the sampling system were stored at -5°C for analysis.

Combustion Residue Analysis

The glass transfer tubes, glass fiber filters, and PUFs were Soxhlet extracted with ethyl acetate and concentrated on a rotary evaporator. Lindane and chlorpyrifos were quantified on a gas chromatograph as previously described. Pesticide recovery (\pm one standard deviation) from spiked PUF and filter samples containing smoke condensate were: lindane — $89\% \pm 9$, $96\% \pm 5$; chlorpyrifos — $101\% \pm 3$, $108\% \pm 8$. Particulate matter was determined by gravimetric analysis of the glass fiber filter.

RESULTS AND DISCUSSION

Insecticide Residues in Treated Trees

Insecticide residues were found at 4, 8, and 12 months in pine wood samples taken from treated trees at levels ranging from 0.32 to 35.8 mg/kg for lindane and < 0.10 to 76.1 mg/kg for chlorpyrifos (Table 1). The high mean lindane concentration (17.2 mg/kg) at 8 months after application decreased to 3.82 mg/kg after 12 months.

Table 1. Mean insecticide residues (mg/kg) in pine firewood 4, 8, and 12 months after application, Ocala National Forest, 1982 - 1983.*

Common name	Months from application		
	4 months	8 months	12 months
	mg/kg		
Lindane	16.9 ± 15.9	17.2 ± 8.30	3.82 ± 3.2
Chlorpyrifos	24.7 ± 11.0	28.8 ± 8.04	40.8 ± 25.4

*Values are means of 5 samples ± 1 standard deviation. Values are on dry weight basis and not segregated by diameter at breast height class.

In agreement with Brady et al. (1980), chlorpyrifos was more persistent in wood than lindane. Residues increased from 24.7 mg/kg at 4 months to 40.8 mg/kg at 12 months; possibly due to residues washing down the tree boles. Berisford et al. (1981a,b) found that chlorpyrifos was very persistent on conifer bark, provided it was not washed off the bark before it dried. The half-life of chlorpyrifos in the field is difficult to ascertain because of the variability in bark coverage and local environmental conditions. Berisford et al. (1981a,b) reported half-lives of approximately 1, 3, 4, and 8 months for 1% or 2% aqueous chlorpyrifos sprays on bark. They also found bark residues as high as 20 - 32% (actual concentrations of 648 - 2403 mg/kg) of the initially applied amount of either 1% or 2% chlorpyrifos on the bark of standing loblolly pines one year post-spray.

Lindane residues on treated pines have been intensively studied (Mizell et al. 1981; Berisford and Brady 1976; Page 1983; Berisford et al. 1981a,b). Lindane persists on pine bark, providing protection from bark beetle attack for up to 6 months after application (Berisford and Brady 1976). Lindane residue levels reported by other authors (Berisford and Brady 1976; Page 1983) are for bark samples only and thus are higher than those reported in Table 1. The chainsaw kerf sampling method produces much more xylem tissue per sample than do the bark discs frequently used in studying insecticide efficacy.

Insecticide Residues in Combustion Products

Insecticide residues found in the slow and rapid combustion runs with the tube furnace are shown in Table 2. As expected, higher recoveries were found under slow burning conditions where distillation of the parent compound can occur. Rapid heating (insertion at 500°C) produced mixed results yielding over 40% recovery of lindane, whereas chlorpyrifos residues dropped from 28 to 4%. Upon examination of the particulate yield for these experiments (10.4%) it was recognized that the 500° C furnace setting was marginal for rapid combustion of the treated wood samples. For evidence of rapid combustion, particulate yields should be

Table 2. Insecticide and particulate yields in smoke condensate from treated wood burned in a tube furnace (average of three runs \pm 1 standard deviation).

Insecticide and furnace conditions	Treatment level mg/kg	Insecticide recovered %	Particulate yield
Lindane			
500" c Slow*	381 \pm 6	43.0 \pm 8.2	11.0 \pm 0.5
500° C Rapid?	383 \pm 4	41.0 \pm 4.0	10.0 \pm 1.0
500" C Rapid† (Repeat)	280-t 9	76.0 \pm 29.0	7.1 \pm 4.0
600" C Rapid†	273 \pm 10	N.D.‡	0.5 \pm 0.1
Chlorpyrifos			
500" c Slow*	504 \pm 7	28.0 \pm 4.9	11.0 \pm 0.5
500" C Rapid†	508 \pm 6	4.2 \pm 3.4	10.0 \pm 1.0
500" C Rapid† (Repeat)	514 \pm 17	2.5 \pm 4.4	7.1 \pm 4.0
600" C Rapid†	501 \pm 18	N.D.‡	0.5 \pm 0.1

*Heating rate 20°C per min from ambient temperature to 500°C.

† Insertion into furnace at temperature indicated.

‡ Not detected at 0.1% carryover level.

approximately 1%. Similar yields were reported by Knight (1983) for wood stoves with open dampers and by Dasch (1982) from open fireplaces. For slow combustion, we were seeking particulate yields of approximately 10%. Similar yields were reported by Knight (1983) for wood burned in stoves with closed dampers.

Additional runs at 500 and 600°C confirmed the marginal status of the 500°C setting for rapid combustion. As shown in Table 2, repeated runs at 500°C continued to produce high levels of lindane (76%) and particulate matter (7%) while runs at 600°C yielded particulate levels below 1% and no detectable insecticide residues.

Potential Exposure Resulting from Burning Treated Wood

An estimate of possible exposure to residues resulting from burning wood treated with chlorpyrifos or lindane can be developed from the data discussed in this paper (Table 3). A sample calculation for slow combustion of lindane would then be as follows:

$$\begin{array}{ccccccc} \text{Insecticide} & & & \text{Stack/room} & \text{Household} & & \text{Room} \\ \text{Burn rate} & \text{conc.} & \text{Carryover} & \text{emission} & \text{volume} & & \text{concentration} \\ 72 \text{ kg/day} \times 25.5 \text{ mg/kg} \times 0.509 \times 10^{-4} & \times 1/350 \text{ m}^3 & = 2.7 \times 10^{-4} \text{ mg/m}^3 \end{array}$$

Wood consumption rates of 3.0 kg/hr for wood stoves and 8.5 kg/hr for fireplaces (DeAngelis et al. 1980b) corresponding to slow and rapid combustion in the tube furnace were used. Insecticidal carryover concentrations were obtained from Table 1 and 2 and correspond to the highest mean concentration plus one standard deviation.

Table 3. Calculation of home exposure resulting from burning lindane or chlorpyrifos treated firewood.

ASSUMPTIONS

Combustion rate:

Slow - 3.0 kg wood/hr or 72 kg/day

Fast- 8.5 kg wood/hr or 104 kg/day

*Ratio of room to stack components:*Conservative value 10^{-4} was used*Pesticide concentration* from Table 1:

Lindane 25.5 mg/kg*

Chlorpyrifos 66.2 mg/kg*

% carryover in smoke from Table 2:

Lindane slow 50.9%*

rapid 0.1%*

Chlorpyrifos slow 33.2%*

rapid 0.1%*

Volume of standard house: 350 m³. For simplicity in calculation it is assumed that ventilation or air exchange did not occur.

Air concentration:

$$CA = (R \times CW \times CO \times E) / V$$

Equation 1

Where:

R = Combustion rate, kg/day

CW = Pesticide concentration in wood mg/kg

CO = Fraction of pesticide carryover in smoke condensate

E = Fraction of flue gases escaping in the room

V = Volume of house (M)

CA = Average daily air concentration of pesticide (mg/m)

 *Highest mean concentration + standard deviation.

An estimate of the room to stack concentrations was difficult to obtain from published literature. Hubble and Harkness (1981) and DeAngelis et al. (1980a) report carbon monoxide stack concentrations between 10,000 - 30,000 ppm for a wide variety of wood burning conditions. In a separate study which examined the effects of wood combustion on residential indoor air quality, Moschandreas and Zabransky (1981) report carbon monoxide home concentrations to be elevated by approximately 1 ppm as a result of wood burning in wood stoves and fireplaces in a residential suburban neighborhood giving a ratio of 1:10,000 or 10^{-4} . In this case, home ventilation and recirculation from neighbor homes, etc., is factored into the ratio. In the case of burning insecticide treated wood, it is unlikely that the entire community would be doing it, so 10^{-4} generates an extreme worst case factor and if in error, is strongly "safe sided."

The Threshold Limit Values (TLV) for lindane and chlorpyrifos are 0.5 mg/m^3 and 0.2 mg/m^3 , respectively (ACGIH, 1984). The TLV is an 8-hour time weighted air quality standard applied to environments where pesticides are manufactured. To convert occupational TLV limits to general population limits, a safety factor of 1/300 has been applied. This is consistent with methodology used by the Environmental Protection Agency and several states to estimate risk from non-criteria pollutants (DeAngelis et al. 1980b). The use of "safe-sided" TLVs helps to compensate for inherent individual human differences in severity of reactions to pesticides.

The safe sided lindane TLV, therefore, is equal to $1/300 \times 0.5 \text{ mg/m}^3$ or 16.7×10^{-4} . Thus the worst case room concentration at $2.7 \times 10^{-4} \text{ mg/m}^3$ for lindane under slow smoldering combustion conditions is only 1/6 of the safe sided TLV. For chlorpyrifos burned under these conditions, the room air concentration of 4.5×10^{-4} is 67% of the safe sided TLV (Table 4). Under conditions of rapid combustion, it is unlikely that insecticide concentrations would be detected in the room since rapid combustion at 600°C results in complete thermal decomposition and thus no detectable pesticide concentration.

Table 4. Average daily room concentrations of lindane or chlorpyrifos from burning pesticide treated wood.

	Combustion rate		"Safe sided" TLV
	Slow	Fast (600°C)	
	$\text{----- } 10^{-4} \text{ mg/m}^3 \text{ -----}$		
Lindane	2.7	0.014	16.7
Chlorpyrifos	4.5	0.038	5.0

The World Health Organization has established an acceptable human daily intake (ADI) of 0.01 mg/kg for chlorpyrifos and/or lindane (Berisford and Cowie 1985). If we assume that a 70 kg adult would inhale $8.6 \text{ m}^3/\text{day}$ (Ganong 1979) and 100% of the pesticide is retained by the body, then average air concentrations generated by slow combustion of firewood would result in systemic absorption of $7.65 \times 10^{-4} \text{ mg/day}$ for lindane and $12.45 \times 10^{-4} \text{ mg/day}$ for chlorpyrifos. The inhalation of lindane and/or chlorpyrifos under these conditions would result in 0.11 and 0.17%, respectively, of the acceptable daily intake.

Lindane residues, like those from many chlorinated hydrocarbons, have been found in human fat and milk (Egan et al. 1965; Hayes 1966; Abbott et al. 1968) indicating it can be absorbed and stored by the body in small quantities. Lindane (O'Brien 1967) and chlorpyrifos ($t_{1/2} = 27 \text{ hrs}$ Nolan et al. 1984) are readily metabolized in the liver and excreted in the urine. It is very unlikely that exposure at the $1 \cdot 10$ part per trillion level predicted in this study would exceed the ability of the human system to metabolize and excrete either pesticide.

CONCLUSIONS

Burning trees that have been treated with chlorpyrifos or lindane at the rates recommended for bark beetle control can, under certain combustion conditions, result in carryover of insecticidal residues. However, even conservative TLVs are

not exceeded when these materials are burned. During slow combustion, the amount of pesticide present in the smoke stream depends on the physical and thermal properties of the parent compound. Stable compounds such as lindane may, under slow burning conditions, have significant (ca. 50%) carryover. Under the same conditions, less stable compounds such as chlorpyrifos will produce markedly less carryover (ca. 33%).

Combustion under rapid flaming conditions with high (600°C) temperatures causes complete decomposition of both lindane and chlorpyrifos. Calculations based on exposure values higher than likely indicate that humans exposed to lindane or chlorpyrifos as a result of residential wood burning should be exposed to levels which are considerably lower than either adjusted occupational exposure limits or acceptable daily intakes. However, because of the **uncertainty** of temperatures reached in any given fireplace or wood burning heater, wood treated with lindane or chlorpyrifos should only be used when a hot, rapidly flaming fire is present. Wood treated with these insecticides should not be used as kindling, or added to a smoldering, less than well-developed fire. Wood treated with these materials should be stored outside prior to burning.

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